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Dec 10, 1992

DERWENT-ACC-NO: 1993-030475

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TITLE: Safe and effective treating agent for hair setting - contains soluble cpd. based on pyridine, oxazine, thiazine, pyridazine, pyrazine, triazine or tetrazine cpds. as component for crosslinking keratin molecules

PATENT-ASSIGNEE:

ASSIGNEE

CODE

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NIBIN

PRIORITY-DATA: 1991JP-0155941 (May 31, 1991)

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ABSTRACTED-PUB-NO: JP 04356411A

BASIC-ABSTRACT:

Agent contains a soluble cpd. having a nitrogen-contg. six-membered ring subst. with at least two halogen atoms as the ingredient for crosslinking keratin molecules. Pref., the agent consists of a first liq. contg. the cpd. having the six-membered ring and second liq. contg. an alkali.

Pref., nitrogen-contg. six-membered ring structures include pyridine, oxazine, thiazine, pyridazine, pyrazine, triazine and tetrazine rings. The cpds. are pref. soluble in water, alcohols and/or other organic solvents. Cpds. include 3-bromo-5-fluoropyridine, 2,6-dichloro-3-phenylpyridine, 3,5-dichloro-2-ethoxypyridine,

2,4-dichloropyrimidine, 2,6-dichloro-5-ethoxy-pyridine, 2,4,6-trichloropyrimidine, 5-bromo-2,6-dichloropyrimidine, 4,6-dichloro-2-methyl mercaptopyrimidine, 3,6-dichloropyridazine, 2,4,6-trichlorotriazine, 2,4-dichloro-6-phenyl triazine and N-(3,5-dichloro-2,4,6-triazinyl- (1)) -aniline.

USE/ADVANTAGE - The agent permits effective setting of the hair in the form of waves or straight without hair damage.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: SAFE EFFECT TREAT AGENT HAIR SET CONTAIN SOLUBLE COMPOUND BASED  
PYRIDINE OXAZINE THIAZINE PYRIDAZINE PYRAZINE TRIAZINE TETRAZINE COMPOUND COMPONENT  
CROSSLINK KERATIN MOLECULAR

DERWENT-CLASS: D21 E13

CPI-CODES: D08-B05; E07-H04;

CHEMICAL-CODES:

Chemical Indexing M3 \*01\*

Fragmentation Code

F012 F013 F014 F015 F016 F431 F541 F551 F580 G010  
G100 H102 H121 H321 H521 H592 H6 H602 H603 H607  
H608 H609 H622 H623 L922 L941 M113 M123 M143 M210  
M211 M212 M271 M272 M280 M281 M320 M413 M510 M521  
M530 M531 M540 M781 M903 M904 Q252

Ring Index

00212

Markush Compounds

199304-B9501-U

Chemical Indexing M3 \*02\*

Fragmentation Code

F013 F015 F431 H6 H601 H603 H622 M280 M320 M413  
M510 M521 M530 M540 M781 M903 M904 Q252

Markush Compounds

199304-B9502-U

SECONDARY-ACC-NO:

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Full	Title	Citation	Front	Review	Classification	Date	Reference			Claims	RMK	Draw D
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7/11

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最終頁に続く

(54) 【発明の名称】 毛髪セット用処理剤

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(57) 【特許請求の範囲】

【請求項1】 カルシウム又はジルコニウムの水溶性化合物0.1～1.0重量%、尿素5～30重量%、システイン0.1～5重量%、オキシカルボン酸1～5重量%を含み、アルカノールアミンによりpH9.0～1

1.0に調整された水溶液から成る第一処理剤、2個以上のハロゲン原子で置換されたトリアジン環をもつ可溶性化合物0.1～3重量%を含む溶液から成る第二処理剤及びアルカリ含有溶液から成る第三処理剤を組み合わせたことを特徴とする毛髪セット用処理剤。

【発明の詳細な説明】

【0001】

【産業上の利用分野】 本発明は、毛髪を損傷することなく、効果的にヘアウエーブ又はヘアストレートのようなセット加工を施すことができる新規な毛髪セット用処

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理剤に関するものである。さらに詳しくいえば、本発明は、毛髪を構成するケラチン分子間をトリアジン化合物で架橋することにより、毛髪のセット加工を行うという全く新しい原理に基づく、毛髪セット用処理剤に関するものである。

【0002】

【従来の技術】 従来、コールドパーマ法としては、チオグリコール酸を主剤とする第一液で毛髪中のシスチン部分のジスルフィド結合を開裂し、所要のセットを施したのち、過酸化水素や臭素酸塩を主剤とする第二液で酸化して再びジスルフィド結合を形成する方法が最も一般的に行われている。

【0003】 しかしながら、この方法においては、毛髪を構成するケラチン分子を化学的に切断するために、かなり強力な処理剤を用いることが必要であり、必然的に

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毛髪の損傷を伴うのを免れず、場合によっては断毛や枝毛を生じるという欠点がある。

【0004】このような損傷を伴う化学反応に基づくコールドパーマ法の欠点を改善するため、ケラチン分子間に金属キレートを形成させてセット加工を施す方法が提案されている（特公昭53-1344号公報）。この方法は、チオグリコール酸と臭素酸塩とを用いる、それまでのコールドパーマ法のように毛髪の損傷を生じることなく、むしろ強度は向上するという利点はあるが、穏和な処理剤を用いているため、セット加工に長時間を有する上に金属キレートの結合力を利用したものであるため、化学結合を利用したもの比べ持続性を欠くのを免れない。

【0005】

【発明が解決しようとする課題】本発明は、従来の毛髪セット処理剤のもつ欠点を克服し、毛髪の損傷を伴うことなく取り扱いが容易で、しかも短時間の簡単な処理で長期間にわたって毛髪のセットを持続させる新規な毛髪セット用処理剤を提供することを目的としてなされたものである。

【0006】

【課題を解決するための手段】本発明者らは、従来のコールドパーマ法の欠点を改善するために、それとは全く異なった原理に基づく毛髪セット法を開発すべく鋭意研究を重ねた結果、ケラチン分子間に金属キレートを形成させる代りにある種の含窒素複素環化合物による化学的な架橋を形成させることによりその目的を達成しうることを見出し、本発明をなすに至った。

【0007】すなわち、本発明は、カルシウム又はジルコニウムの水溶性化合物0.1～1.0重量%、尿素5～30重量%、システイン0.1～5重量%、オキシカルボン酸1～5重量%を含み、アルカノールアミンによりpH9.0～11.0に調整された水溶液から成る第一処理剤、2個以上のハロゲン原子で置換されたトリアジン環をもつ可溶性化合物0.1～3重量%を含む溶液から成る第二処理剤及びアルカリ含有溶液から成る第三処理剤を組み合わせたことを特徴とする毛髪セット用処理剤を提供するものである。

【0008】本発明の第一処理剤は、カルシウム又はジルコニウムの水溶性化合物0.1～1.0重量%、尿素5～30重量%、システイン0.1～5重量%、オキシカルボン酸1～5重量%を含み、アルカノールアミンによりpH9.0～11.0に調整された水溶液から成

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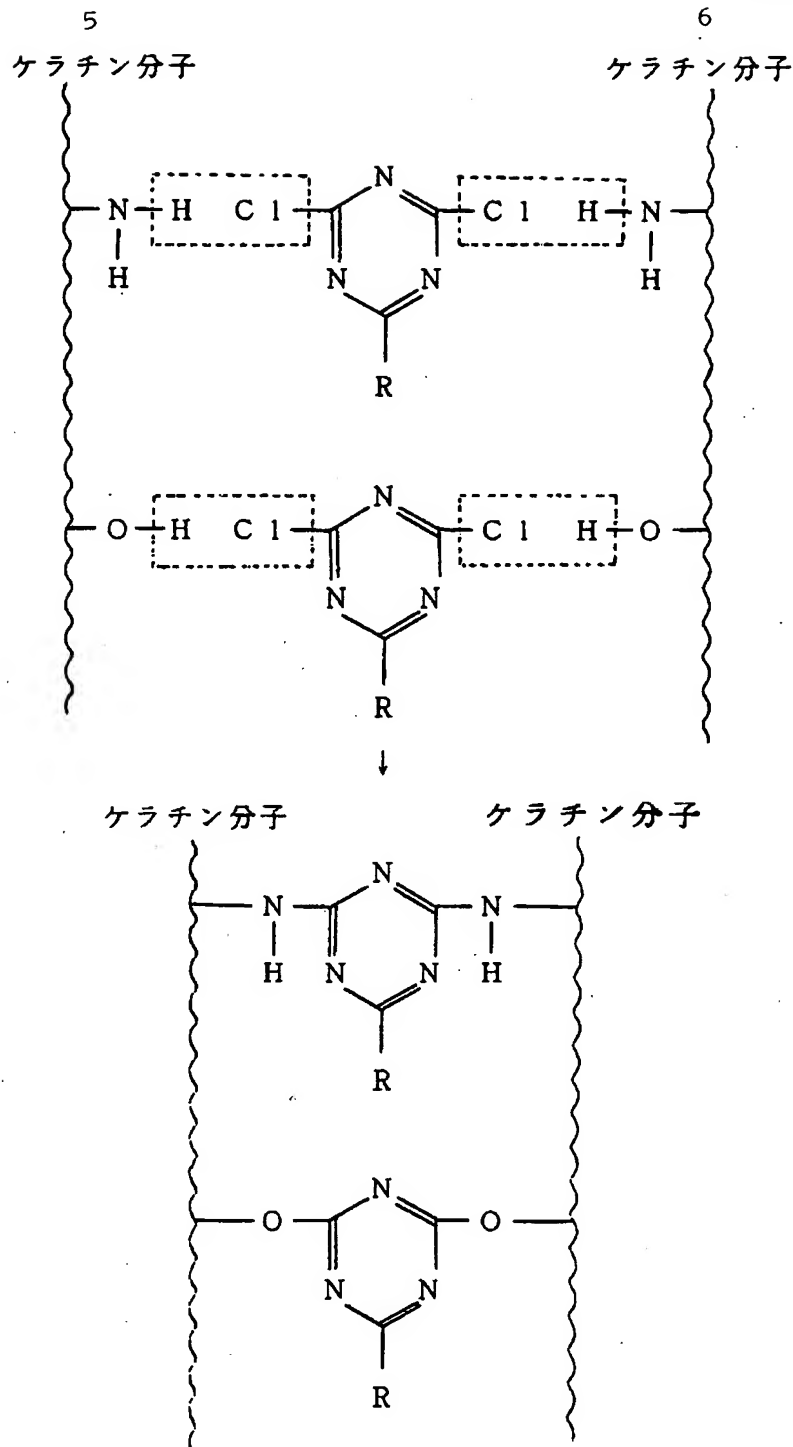
る。本発明の第二処理剤において、主要活性成分として用いられる化合物は、2個以上のハロゲン原子で置換されたトリアジン環をもつ可溶性化合物であることが必要である。このような化合物の例としては、水又は水性溶媒に可溶なジ又はトリハロゲン化トリアジン化合物、例えば2,4,6-トリクロロトリアジン、2-フェニルアミノ-4,6-ジクロロトリアジン、2,4-ジクロロ-6-フェニルトリアジン、N-(3,5-ジクロロ-2,4,6-トリアジニル-(1))-アニリンなどを挙げることができる。

【0009】また、白髪や脱色毛の場合、セット加工と同時に染毛処理を施すことがあるが、この場合には、前記した2個以上のハロゲン原子で置換されたトリアジン環をもつ反応染料を用いるのが有利である。このような反応染料としては、ジクロロトリアジン型反応性染料、例えばプロシオン・スカーレットRS、プロシオン・ブリリヤントレッドB、5BS、8BS、プロシオン・ブリリヤント・オレンジGS、2RS、プロシオン・イエローRS、GRS、4RS、8GS、プロシオン・ブリリヤント・ブルーRS、プロシオン・バイオレット3R、プロシオン・ブルー2R、2GS、プロシオン・グリーン2BS、プロシオン・ブラックPN、プロシオン・グレイPNRなどを挙げるができる。

【0010】そのほか、シー・エル・バード及びダブリュ・エス・ボストン（C. L. Bird and W. S. Boston）編、「セオリー・オブ・カラーレイション・オブ・テキスタイルズ（Theory of Coloration of Textiles）」第326～357ページのピー・ライズ（P. Rys）及びエイチ・ゾリンガー（H. Zollinger）の報文に示されている反応性染料のうち、3,5-ジクロロ-2,4,6-トリアジニル基を含有しているものも好適に用いることができる。

【0011】この2個以上のハロゲン原子で置換されたトリアジン環をもつ可溶性化合物は、毛髪を構成しているケラチン分子中の水酸基、チオール基、アミノ基、カルボキシル基と反応して、ケラチン分子間で架橋を形成するためのものである。これを、2,4-ジクロロ-1,3,5-トリアジニル基をもつ化合物とケラチン分子中のアミノ基との反応を例として説明すると以下に反応式に示すような経過で架橋が形成される。

【化1】



(ただし、Rは有機残基)

【0012】そして、このような架橋を形成するために、ケラチン分子間に介挿するためには、トリアジン環の大きさが最も適しており、それ以外の六員環や五員環では、上記したような完全な架橋形成が行われない。また、六員環上の2個のハロゲン原子の相対的な結合位置としては、オルト位置、パラ位置のものでもよいが、\*50

\*の結合距離の点を考慮すると、特にメタ位置のものが有利である。

【0013】本発明の第二処理剤は、このようなトリアジン環をもつ可溶性化合物を0.1～3重量%の濃度になるように溶剤に溶解することによって調製される。この際の溶剤としては、水が好ましいが、トリアジン環をもつ可溶性化合物が水に溶解しにくい場合には、アルコ

ール、アセトン、ジメチルホルムアルデヒド、ジメチルスルホキシドのような有機溶剤に溶かしたのち、水で希釈することもできる。

【0014】本発明の第二処理剤中には、前記の活性成分のほかに、毛髪と有効成分との親和性を増大させるための界面活性剤や毛髪に対するぬれ性を改善するためのグリコール、ジエチレングリコール、エチルセロソルブのような湿潤助剤、尿素のような活性化助剤、亜硫酸ナトリウム、硫酸ナトリウムのような塩類、安定剤、分散剤、香料など、毛髪セット用処理剤に慣用されている添加成分を含有させることができる。

【0015】本発明の毛髪セット用処理剤を用いてヘアウェーブを施すには、ロッドを用いてカール加工を施した毛髪を第一及び第二処理剤に浸せきするか、あるいは処理剤に浸せきした毛髪にカール加工を施したのち、第三処理剤すなわちアルカリ含有溶液でこの毛髪を処理して、トリアジン環をもつ化合物とケラチン分子との間の反応を行わせ、カールを固定させる。この際に用いる第三処理剤のアルカリ含有溶液としては、炭酸ナトリウム、炭酸カリウム、炭酸水素ナトリウム、炭酸水素カリウム、酢酸ナトリウム、酢酸カリウムなどのアルカリ金属塩やメチルアミン、エチルアミン、ジメチルアミン、ジエチルアミン、トリメチルアミン、トリエチルアミン、エタノールアミン、ジエタノールアミン、トリエタノールアミンなどの有機塩基の溶液が用いられる。この溶液の溶媒としては、水が好ましいが、そのほか、アルコール、アセトン、ジメチルホルムアルデヒドなどの有機溶剤や、これと水との混合溶剤も用いることができる。

【0016】このアルカリ溶液中のアルカリの濃度は、0.1～5重量%、好ましくは0.3～2重量%の範囲内で選ばれる。また、このアルカリ溶液のpHとしては9～11が適当である。

【0017】本発明の処理剤によるケラチン分子間の架橋形成反応は、常温でも円滑に進行するが、反応を促進するために、30～50℃の温度に加温することもできる。

【0018】また、本発明者らは、先に毛髪のセット加工を効果的に行うための前処理剤として、カルシウム又はジルコニウムの水溶性化合物0.1～1.0重量%、尿素5～30重量%、システイン0.1～5重量%、オキシカルボン酸1～5重量%を含み、アルカノールアミンによりpH9.0～11.0に調整された水溶液から成る処理剤を開発したが(特願平2-272342号)、本発明の毛髪セット用処理剤は、この前処理剤と組み合わせて使用することにより、いっそう良好な結果を得ることができる。

【0019】

【実施例】次に、実施例により本発明をさらに詳細に説明する。なお、各例における物性試験は以下のように行った。

【0020】(1) ウェーブ効果測定試験；

長さ18cmの女性正常毛約50本を用いて毛束を作る。この毛束を直径6mm骨ロッドに5巻きしてゴムで固定し、所定の毛髪セット加工処理を施したのち、ロッドから取りはずし、水洗いして自然乾燥する。乾燥したのちセット状態を図1に示すように分類し、6段階(0級ないし5級)で評価する。

【0021】(2) 強度試験；

ウェーブ効果測定試験の場合と同様にして毛髪セット加工を施した毛束を、ロッドに巻いたまま十分に水洗し、自然乾燥する。次に、この毛束をロッドから取りはずし、その中からランダム20本の毛髪を取り出し、簡易毛髪強度計(レディーバード社製)を用いて、それぞれの毛髪の太さ及び強度を測定する。測定条件としては室温(24～26℃)、湿度50%を用いた。毛髪の太さは3か所で測定し、それらの平均値をmmで表わし、また強度は毛髪に荷重を加え、切断したときの数値(g)をもって表わした。

【0022】処方例1

塩化ジルコニル( $ZrOCl_2 \cdot 8H_2O$ ) 0.5重量部、尿素5重量部、L-システイン3.0重量部及びクエン酸3.0重量部を精製水に溶解して全量を100重量部とし、モノエタノールアミンをpH10.0になるまで添加することによりA液を調製した。別にポリオキシエチレンオレイルエーテル1.0重量部及び無水亜硫酸ナトリウム2.0重量部を精製水に溶解して全量を100重量部とすることによりB液を調製した。次いで、このA液とB液とを等量ずつ混合することによりpH9.5の第一処理剤を得た。

【0023】処方例2

塩化ジルコニル0.5重量部、尿素10.0重量部、クエン酸3.0重量部を精製水100重量部に溶解した溶液中に、システインを0%、0.5%、1%、3%、5%及び7%の濃度になるように添加したのち、モノエタノールアミンによりpH10.0に調整し、A液を調製した。別に、ポリオキシエチレンオレイルエーテル1.0重量部及び無水亜硫酸ナトリウム2.0重量部を精製水に溶解して全量を100重量部とすることによりB液を調製した。次いで、このA液とB液とを等量ずつ混合することによりpH9.5のシステイン含有量の異なる第一処理剤6種を得た。

【0024】処方例3

塩化シアヌルをエタノールに溶解し、濃度0.1重量%、0.3重量%、0.5重量%、1.0重量%及び2.0重量%の5種の第二処理剤を調製する。

【0025】処方例4

水100ml当り、ジクロルトリアジニル基をもつ黒色反応性染料(アイ・シー・アイ社製、商品名プロシオン・ブラックPN)2g、硫酸ナトリウム5g及び尿素10gを含む第二処理剤を調製する。

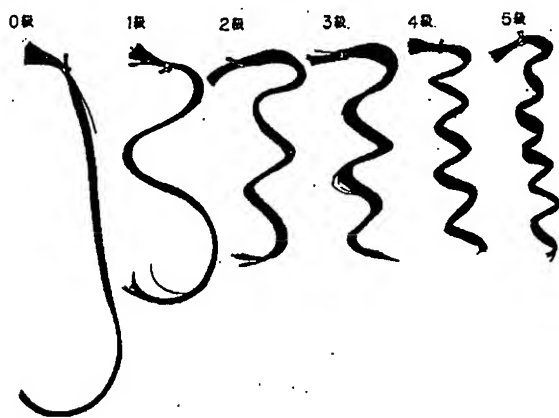
## 【0026】参考例

処方例1で得た第一処理剤により、あらかじめ55℃で10分間処理した試験用毛髪を直径6mmのロッドに巻き、これらの5種の塩化シアヌルエタノール溶液のそれぞれに浸し、25℃で10分間処理する。次いで、これらの試料を取り出したのち、0.1%炭酸ナトリウム水溶液に浸し、25℃で10分間処理し、セットを固定する。このようにしてセット加工した毛髪を水洗し、風乾したのち、そのウェーブ効果を測定し、この結果を図2のグラフIとして示す。また、これらの試料のそれぞれについて強度を測定したところ、5.8~6.0g/dの範囲であった。さらに、塩化シアヌルエタノール溶液と炭酸ナトリウム水溶液による処理温度を40℃に変えること以外は、上記と同じ操作でセット加工を行い、そのウェーブ効果を測定したところ、図2のグラフIIに示す結果が得られた。また、これらの試料の強度は6.1~6.3g/dの範囲であった。

## 【0027】実施例1

処方例1で得た第一処理剤に、ロッドに巻いた白髪まじりの黒色毛髪を巻き付け、37℃で20分間処理したのち、前記のようにして調製した毛髪セット用処理剤に浸せし、25℃で10分間処理する。次いで、この処理剤の中へ炭酸ナトリウム0.5g/100mlを加えて、第三処理剤とし、25℃でさらに10分間処理する。このようにして、セット加工した毛髪を水洗し、風乾したところ、均一な黒色に染色された毛髪が得られた。次に、そのウェーブ効果及び強度を測定したところ、ウェーブ効果は3.5級、強度は5.8g/dであった。また、第一処理剤の処理条件を55℃で10分間に変えること以外は、全く同様に操作して、セット加工

【図1】



したところ同じように均一な黒色に染色された毛髪が得られた。このもののウェーブ効果は5級、強度は6.2g/dであった。

## 【0028】実施例2

処方例2で得た各第一処理剤を用いて白髪まじりの毛髪束を37℃で20分間又は55℃で10分間処理したのち、これをロッドに巻いてカールさせ、実施例1と同じ毛髪セット用処理剤に浸せし、同じようにしてセット加工した。このようにしていずれも均一な黒色に染色された毛髪を得た。これらの毛髪における、第一処理剤中のシステイン濃度とウェーブ効果との関係及びシステイン濃度と強度との関係をグラフとして図3及び図4に示す。図中Iは37℃で20分間処理したもの、IIは55℃で10分間処理したものである。これらの図から分かるように、本発明の処理剤を用いて毛髪のセット加工を行う場合には、第一処理剤の組成を適当に選ぶことによって、よりすぐれたウェーブ効果及び強度を得ることができる。

## 【図面の簡単な説明】

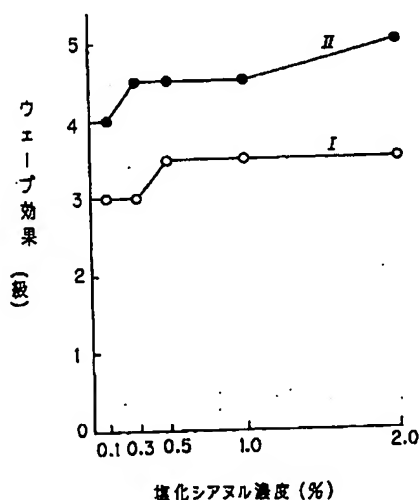
【図1】 本発明におけるウェーブ効果の評価段階を示す毛髪の状態図。

【図2】 本発明の毛髪セット用処理剤中の有効成分濃度とウェーブ効果との関係を示すグラフ。

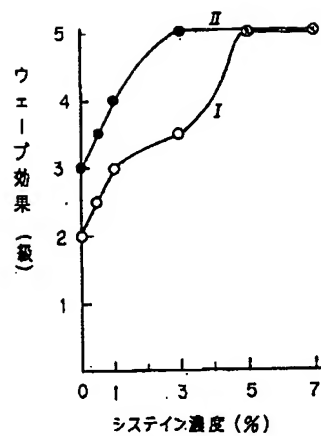
【図3】 本発明の毛髪セット用処理剤の第一処理剤におけるシステイン濃度とウェーブ効果の関係を示すグラフ。

【図4】 本発明の毛髪セット処理剤の第一処理剤におけるシステイン濃度と処理毛髪の強度との関係を示すグラフ。

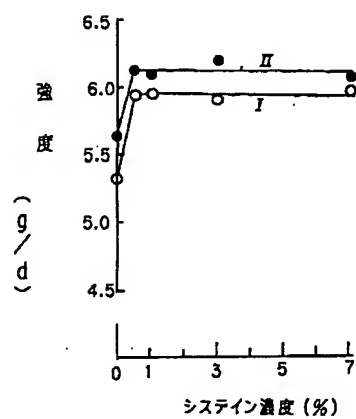
【図2】



【図3】



【図4】



フロントページの続き

(58)調査した分野(Int.Cl.<sup>7</sup>, DB名)

A61K 7/09

A61K 7/11



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CLAIMS

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(57) [Claim(s)]

[Claim 1] Calcium or 0.1 - 1.0 % of the weight of water-soluble compounds of a zirconium, 5 - 30 % of the weight of ureas, 0.1 - 5 % of the weight of cystines, and 1 - 5 % of the weight of hydroxy acid are included. The first processing agent which consists of the water solution adjusted to pH 9.0-11.0 by alkanolamine, The processing agent for a hair set characterized by combining the third processing agent which consists of the second processing agent and alkali content solution which consist of the solution containing 0.1 - 3 % of the weight of fusibility compounds with the triazine ring permuted by two or more halogen atoms.

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the new processing agent for a hair set which can perform hair wave or set processing like a hair straight effectively, without damaging hair. If it says in more detail, this invention relates to the processing agent for a hair set based on the completely new principle of performing set processing of hair, by constructing a bridge with a triazine compound in between the keratin molecules which constitute hair.

[0002]

[Description of the Prior Art] After cleaving the disulfide bond of the cystine part in hair as a cold wave method with the first liquid which uses thioglycolic acid as base resin and giving a necessary set conventionally, most generally the approach of oxidizing with the second liquid which uses a hydrogen peroxide and bromate as base resin, and forming a disulfide bond again is performed.

[0003] However, in this approach, in order to cut chemically the keratin molecule which constitutes hair, it is required to use a quite powerful processing agent, and it does not escape being inevitably accompanied by damage on hair, but there is a fault of producing a broken hair and split hair depending on the case.

[0004] In order to improve the fault of the cold wave method based on the chemical reaction accompanied by such damage, the method of making a metal chelate form between keratin molecules, and performing set processing is proposed (JP,53-1344,B). Although there is an advantage that reinforcement improves, rather, without this approach producing damage on hair like the cold wave method till then for using thioglycolic acid and bromate, in order to have long duration upwards in set processing since the mild processing agent is used, and to use the bonding strength of a metal chelate, it does not escape lacking durability compared with the thing using a chemical bond.

[0005]

[Problem(s) to be Solved by the Invention] It is made for the purpose of offering the new processing agent for a hair set in which handling is easy an agent, is made to maintain the set of hair over a long period of time by processing with a short time easy moreover, without this invention's conquering the fault which the conventional hair set processing agent has, and being accompanied by damage on hair, and it deals.

[0006]

[Means for Solving the Problem] this invention persons came to make a header and this invention for the ability of the purpose to be attained by making the chemical bridge formation by the nitrogen-containing heterocyclic compound of a certain kind form instead of making a metal chelate form between keratin molecules, as a result of repeating research wholeheartedly that the hair setting method based on a completely different principle from it should be developed, in order to improve the fault of the conventional cold wave method.

[0007] This invention Namely, calcium or 0.1 - 1.0 % of the weight of water-soluble compounds of a zirconium, 5 - 30 % of the weight of ureas, 0.1 - 5 % of the weight of cystines, and 1 - 5 % of the weight

of hydroxy acid are included. The first processing agent which consists of the water solution adjusted to pH 9.0-11.0 by alkanolamine, The processing agent for a hair set characterized by combining the third processing agent which consists of the second processing agent and alkali content solution which consist of the solution containing 0.1 - 3 % of the weight of fusibility compounds with the triazine ring permuted by two or more halogen atoms is offered.

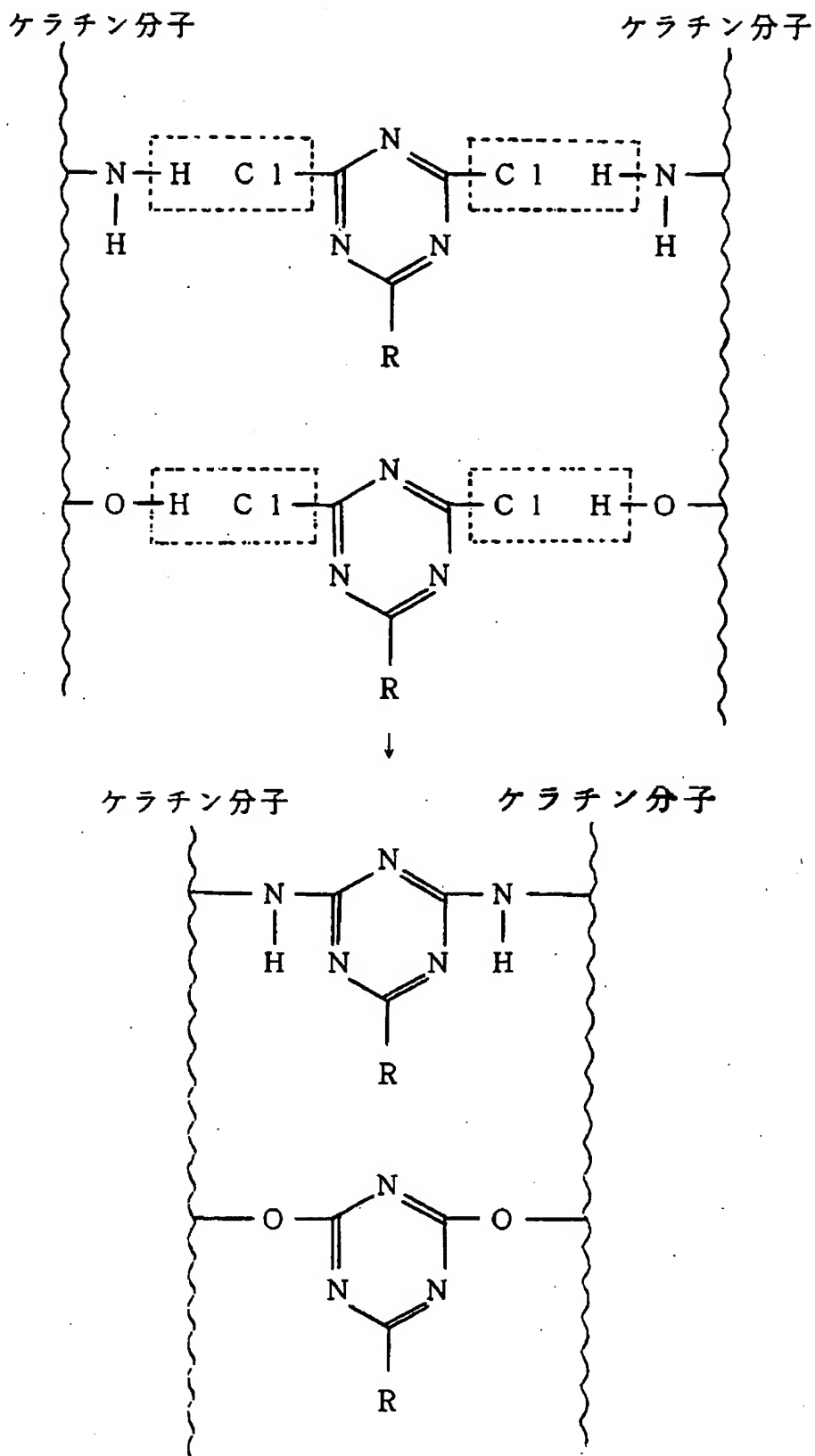
[0008] The first processing agent of this invention consists of the water solution adjusted to pH 9.0-11.0 by alkanolamine including calcium or 0.1 - 1.0 % of the weight of water-soluble compounds of a zirconium, 5 - 30 % of the weight of ureas, 0.1 - 5 % of the weight of cystines, and 1 - 5 % of the weight of hydroxy acid. In the second processing agent of this invention, the compound used as a main active ingredient needs to be a fusibility compound with the triazine ring permuted by two or more halogen atoms. meltable to water or aquosity solvent as example of such compound II or Tori halogenation triazine compound, 2 and 4, 6-TORIKURORO triazine, and 2-phenylamino - 4, 6-dichloro triazine, 2, 4-dichloro-6-phenyl triazine, N-[3, and 5-dichloro - a 2, 4, and 6-thoriadiny1-(1)]-aniline etc. can be mentioned. [ for example, ]

[0009] Moreover, in the case of canities or decolorization hair, hair dyeing processing may be performed to set processing and coincidence, but it is advantageous to use reactive dye with the triazine ring permuted by the two or more above mentioned halogen atoms in this case. As such reactive dye, dichloro triazine mold reactive dye RS, for example, pro Aster tataricus Scarlett The pro Aster tataricus BURIRIYAN tread B, 5BS, 8BS, pro Aster tataricus BURIRIYANTO Orange GS, 2RS, The pro Aster tataricus yellow RS and GRS, 4RS, 8GS, pro Aster tataricus BURIRIYANTO blue RS, Pro Aster tataricus violet 3R, pro Aster tataricus blue 2R, 2GS, pro Aster tataricus Green 2BS, the pro Aster tataricus black PN, the pro Aster tataricus gray PNR, etc. can be mentioned.

[0010] In addition, the volume C El Byrd and on W S Boston (C. L.Bird and W.S.Boston), "Theory OBU color lei SHON OBU TEKISUTAIRUZU (Theory of Coloration ofTextiles) " Inside of the reactive dye shown in the report of the page [ 326-357th ] Py rise (P. Rys) and EICHI Zollinger (H. Zollinger), 3 and 5-dichloro - the thing containing 2, 4, and 6-thoriadiny1 group can also be used suitably.

[0011] The fusibility compound with the triazine ring permuted by this two or more halogen atom is for reacting with the hydroxyl group in the keratin molecule which constitutes hair, a thiol group, the amino group, and a carboxyl group, and forming bridge formation between keratin molecules. this -- 2 and 4-dichloro - if the reaction of a compound with 1, 3, and 5-thoriadiny1 group and the amino group in a keratin molecule is explained as an example, bridge formation will be formed by progress as shown in a reaction formula below.

[Formula 1]



(However, R organic residue)

[0012] And in order to form such bridge formation and to insert between keratin molecules, the magnitude of a triazine ring is most suitable and perfect arch forming which was described above is not

performed in another six membered ring or another five membered ring. Moreover, as a relative joint location of two halogen atoms on a six membered ring, although the thing of an alt.location and a para position may be used, if the point of the bond distance is taken into consideration, especially the thing of a meta-location is advantageous.

[0013] The second processing agent of this invention is prepared by dissolving a fusibility compound with such a triazine ring in a solvent so that it may become 0.1 - 3% of the weight of concentration. It can also dilute with water, after melting to an organic solvent like alcohol, an acetone, dimethyl formaldehyde, and dimethyl sulfoxide as a solvent in this case in being hard to dissolve a fusibility compound with a triazine ring in water although water is desirable.

[0014] The addition component commonly used by the processing agent for hair sets, such as salts like an activation assistant like the glycol for improving the wettability over the surface active agent and hair for increasing the compatibility of hair and an active principle other than the aforementioned active ingredient, a diethylene glycol, a humid assistant like ethylcellosolve, and a urea, a sodium sulfite, and a sodium sulfate, a stabilizer, a dispersant, and perfume, can be made to contain in the second processing agent of this invention.

[0015] In order to give a hair wave using the processing agent for a hair set of this invention, after performing curl processing to the hair which carried out the dipping of the hair which performed curl processing using the rod to the first and second processing agent, or carried out the dipping to the processing agent, process this hair in the third processing agent, i.e., an alkali content solution, make the reaction between the compounds and the keratin molecules have a triazine ring perform, and curl makes fix. In this case, as an alkali content solution of the third processing agent to be used, the solution of organic bases, such as alkali-metal salts, such as a sodium carbonate, potassium carbonate, a sodium hydrogencarbonate, a potassium hydrogencarbonate, sodium acetate, and potassium acetate, monomethylamine, ethylamine, dimethylamine, diethylamine, a trimethylamine, triethylamine, ethanolamine, diethanolamine, and triethanolamine, is used. As a solvent of this solution, although water is desirable, organic solvents, such as alcohol, an acetone, and dimethylformamide, and the partially aromatic solvent of this and water can also be used.

[0016] The concentration of the alkali in this alkali solution is preferably chosen 0.1 to 5% of the weight by 0.3 - 2% of the weight of within the limits. Moreover, as pH of this alkali solution, 9-11 are suitable.

[0017] Although the arch-forming reaction between the keratin molecules by the processing agent of this invention advances smoothly also in ordinary temperature, in order to promote a reaction, it can also be warmed in temperature of 30-50 degrees C.

[0018] moreover, this invention persons as a pretreatment agent for performing set processing of hair effectively previously Calcium or 0.1 - 1.0 % of the weight of water-soluble compounds of a zirconium, 5 - 30 % of the weight of ureas, 0.1 - 5 % of the weight of cystines, and 1 - 5 % of the weight of hydroxy acid are included. Although the processing agent which consists of the water solution adjusted to pH 9.0-11.0 by alkanolamine was developed (Japanese Patent Application No. No. 272342 [ two to ]), the processing agent for a hair set of this invention can obtain a much more good result by using it combining this pretreatment agent.

[0019]

[Example] Next, an example explains this invention to a detail further. In addition, the physical-properties trial in each example was performed by [ as being the following ].

[0020] (1) The wave effectiveness measurement trial;

A hair-bundle is made using 50 female normal \*\*\*\* with a die length of 18cm. After winding this hair-bundle around the diameter bone rod of 6mm five, using it as it, fixing with rubber and performing predetermined hair set processing processing, it removes, washes in cold water and seasons naturally from a rod. After drying, a set condition is classified as shown in drawing 1 , and it evaluates in six steps (the 0th class thru/or the 5th class).

[0021] (2) Strength test;

Winding around a rod, the hair-bundle which performed hair set processing like the case of the wave effectiveness measurement trial is fully rinsed, and is seasoned naturally. Next, this hair-bundle is

removed from a rod, the hair of 20 random is taken out from that inside, and the size and reinforcement of each hair are measured using a simple hair strong ductilometer (ready bird company make). As a Measuring condition, a room temperature (24-26 degrees C) and 50% of humidity were used. The size of hair was measured by three places, those averages were expressed with mm, and reinforcement was applied to hair and expressed the load with it with the numeric value (g) when cutting.

[0022] The example of formula 1 zirconyl-chloride ( $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ ) 0.5 weight section, the urea 5 weight section, the L-cystine 3.0 weight section, and the citric-acid 3.0 weight section were dissolved in purified water, the whole quantity was made into the 100 weight sections, and A liquid was prepared by adding monoethanolamine until it is set to pH10.0. B liquid was prepared by dissolving the polyoxyethylene oleyl ether 1.0 weight section and the dried-sodium-sulfite 2.0 weight section in purified water independently, and making the whole quantity into the 100 weight sections. Subsequently, the first processing agent of pH9.5 was obtained by mixing this A liquid and B liquid equivalent [ every ].

[0023] In the solution which dissolved the example of formula 2 zirconyl-chloride 0.5 weight section, the urea 10.0 weight section, and the citric-acid 3.0 weight section in the purified water 100 weight section, after adding a cystine so that it may become 0%, 0.5%, 1%, 3%, 5%, and 7% of concentration, monoethanolamine adjusted to pH10.0 and A liquid was prepared. B liquid was prepared by dissolving the polyoxyethylene oleyl ether 1.0 weight section and the dried-sodium-sulfite 2.0 weight section in purified water, and making the whole quantity into the 100 weight sections independently. Subsequently, first processing agent 6 kinds with which the cystine contents of pH9.5 differ were acquired by mixing this A liquid and B liquid equivalent [ every ].

[0024] Example of formula 3 cyanuric chloride is dissolved in ethanol, and five sorts of second processing agents, 0.1 % of the weight of concentration, 0.3 % of the weight, 0.5 % of the weight, 1.0 % of the weight, and 2.0 % of the weight, are prepared.

[0025] The second processing agent containing 2g (made in ICI, Inc., trade name pro Aster tataricus black PN) of black reactive dye, 5g of sodium sulfates, and 10g of ureas per 100ml of example of formula 4 water and with a dichloro thoriadynyl group is prepared.

[0026] By the first processing agent obtained in the example 1 of the example formula of reference, the hair for a trial beforehand processed for 10 minutes at 55 degrees C is wound around a rod with a diameter of 6mm, and it dips in each of five sorts of these cyanuric chloride ethanol solutions, and processes for 10 minutes at 25 degrees C. Subsequently, after taking out these samples, it dips in a sodium-carbonate water solution 0.1%, and processes for 10 minutes at 25 degrees C, and a set is fixed. Thus, the hair which carried out set processing is rinsed, after being air-dry, that wave effectiveness is measured and this result is shown as a graph I of drawing 2. Moreover, when reinforcement was measured about each of these samples, it was the range of 5.8 - 6.0 g/d. Furthermore, except changing the processing temperature by the cyanuric chloride ethanol solution and the sodium-carbonate water solution into 40 degrees C, when set processing was performed by the same actuation as the above and the wave effectiveness was measured, the result shown in the graph II of drawing 2 was obtained. Moreover, the reinforcement of these samples was the range of 6.1 - 6.3 g/d.

[0027] After twisting the grizzled black hair wound around the rod around the first processing agent obtained in the example 1 of example 1 formula and processing for 20 minutes at 37 degrees C, a dipping is carried out to the processing agent for a hair set which is the above, and was made and prepared, and it processes for 10 minutes at 25 degrees C. Subsequently, 0.5g of sodium carbonates and 100ml are added into this processing agent, and it considers as the third processing agent, and processes for 10 more minutes at 25 degrees C. Thus, the hair which carried out set processing was rinsed, and when air-dry, the uniform hair dyed black was obtained. Next, when the wave effectiveness and reinforcement were measured, the number of the wave effectiveness was 3.5 and reinforcement was 5.8 g/d. Moreover, the hair uniform like the place which operated similarly and completely carried out set processing dyed black was obtained except changing the processing conditions of the first processing agent in 10 minutes at 55 degrees C. The number of the wave effectiveness of this thing was five, and reinforcement was 6.2 g/d.

[0028] After processing a grizzled hair bundle for 10 minutes for 20 minutes or at 55 degrees C by 37 degrees C using each first processing agent obtained in the example 2 of example 2 formula, wind this around a rod, it was made to curl, the dipping was carried out to the same processing agent for a hair set as an example 1, and set processing was carried out similarly. Thus, all obtained the uniform hair dyed black. It is shown in drawing 3 and drawing 4 by making relation of the cystine concentration in the first processing agent and the wave effectiveness in such hair, and relation between cystine concentration and reinforcement into a graph. As for the inside I of drawing, what was processed for 20 minutes at 37 degrees C, and II process for 10 minutes at 55 degrees C. As shown in these drawings, when performing set processing of hair using the processing agent of this invention, more excellent the wave effectiveness and reinforcement can be obtained by choosing the presentation of the first processing agent suitably.

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**TECHNICAL FIELD**

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TECHNICAL PROBLEM

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MEANS

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[0008] The first processing agent of this invention consists of the water solution adjusted to pH 9.0-11.0 by alkanolamine including calcium or 0.1 - 1.0 % of the weight of water-soluble compounds of a zirconium, 5 - 30 % of the weight of ureas, 0.1 - 5 % of the weight of cystines, and 1 - 5 % of the weight of hydroxy acid. In the second processing agent of this invention, the compound used as a main active ingredient needs to be a fusibility compound with the triazine ring permuted by two or more halogen atoms. meltable to water or aqueous solvent as example of such compound II or Tori halogenation triazine compound, 2 and 4, 6-TORIKURORO triazine, and 2-phenylamino - 4, 6-dichloro triazine, 2, 4-dichloro-6-phenyl triazine, N-[3, and 5-dichloro - a 2, 4, and 6-thoriadiny1-(1)]-aniline etc. can be mentioned. [ for example, ]

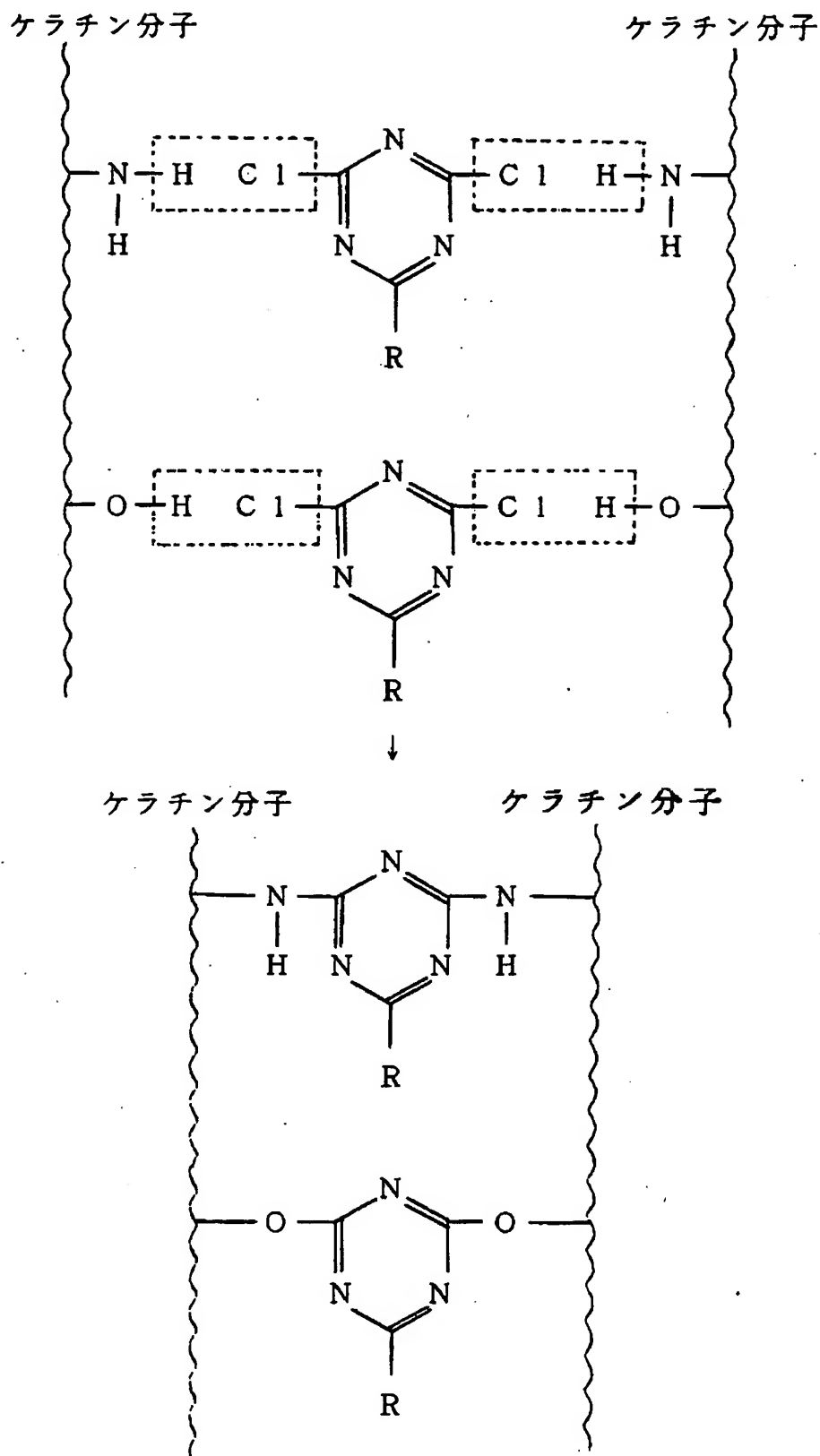
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[Formula 1]



(However, R organic residue)

[0012] And in order to form such bridge formation and to insert between keratin molecules, the magnitude of a triazine ring is most suitable and perfect arch forming which was described above is not performed in another six membered ring or another five membered ring. Moreover, as a relative joint location of two halogen atoms on a six membered ring, although the thing of an alt. location and a para position may be used, if the point of the bond distance is taken into consideration, especially the thing of a meta-location is advantageous.

[0013] The second processing agent of this invention is prepared by dissolving a fusibility compound with such a triazine ring in a solvent so that it may become 0.1 - 3% of the weight of concentration. It can also dilute with water, after melting to an organic solvent like alcohol, an acetone, dimethyl formaldehyde, and dimethyl sulfoxide as a solvent in this case in being hard to dissolve a fusibility compound with a triazine ring in water although water is desirable.

[0014] The addition component commonly used by the processing agent for hair sets, such as salts like an activation assistant like the glycol for improving the wettability over the surface active agent and hair for increasing the compatibility of hair and an active principle other than the aforementioned active ingredient, a diethylene glycol, a humid assistant like ethylcellosolve, and a urea, a sodium sulfite, and a sodium sulfate, a stabilizer, a dispersant, and perfume, can be made to contain in the second processing agent of this invention.

[0015] In order to give a hair wave using the processing agent for a hair set of this invention, after performing curl processing to the hair which carried out the dipping of the hair which performed curl processing using the rod to the first and second processing agent, or carried out the dipping to the processing agent, process this hair in the third processing agent, i.e., an alkali content solution, make the reaction between the compounds and the keratin molecules have a triazine ring perform, and curl makes fix. In this case, as an alkali content solution of the third processing agent to be used, the solution of organic bases, such as alkali-metal salts, such as a sodium carbonate, potassium carbonate, a sodium hydrogencarbonate, a potassium hydrogencarbonate, sodium acetate, and potassium acetate, monomethylamine, ethylamine, dimethylamine, diethylamine, a trimethylamine, triethylamine, ethanolamine, diethanolamine, and triethanolamine, is used. As a solvent of this solution, although water is desirable, organic solvents, such as alcohol, an acetone, and dimethylformamide, and the partially aromatic solvent of this and water can also be used.

[0016] The concentration of the alkali in this alkali solution is preferably chosen 0.1 to 5% of the weight by 0.3 - 2% of the weight of within the limits. Moreover, as pH of this alkali solution, 9-11 are suitable.

[0017] Although the arch-forming reaction between the keratin molecules by the processing agent of this invention advances smoothly also in ordinary temperature, in order to promote a reaction, it can also be warmed in temperature of 30-50 degrees C.

[0018] moreover, this invention persons as a pretreatment agent for performing set processing of hair effectively previously Calcium or 0.1 - 1.0 % of the weight of water-soluble compounds of a zirconium, 5 - 30 % of the weight of ureas, 0.1 - 5 % of the weight of cystines, and 1 - 5 % of the weight of hydroxy acid are included. Although the processing agent which consists of the water solution adjusted to pH 9.0-11.0 by alkanolamine was developed (Japanese Patent Application No. No. 272342 [ two to ]), the processing agent for a hair set of this invention can obtain a much more good result by using it combining this pretreatment agent.

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[Translation done.]

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EXAMPLE

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[Example] Next, an example explains this invention to a detail further. In addition, the physical-properties trial in each example was performed by [ as being the following ].

[0020] (1) The wave effectiveness measurement trial;

A hair-bundle is made using 50 female normal \*\*\*\* with a die length of 18cm. After winding this hair-bundle around the diameter bone rod of 6mm five, using it as it, fixing with rubber and performing predetermined hair set processing processing, it removes, washes in cold water and seasons naturally from a rod. After drying, a set condition is classified as shown in drawing 1 , and it evaluates in six steps (the 0th class thru/or the 5th class).

[0021] (2) Strength test;

Winding around a rod, the hair-bundle which performed hair set processing like the case of the wave effectiveness measurement trial is fully rinsed, and is seasoned naturally. Next, this hair-bundle is removed from a rod, the hair of 20 random is taken out from that inside, and the size and reinforcement of each hair are measured using a simple hair strong ductilometer (ready bird company make). As a Measuring condition, a room temperature (24-26 degrees C) and 50% of humidity were used. The size of hair was measured by three places, those averages were expressed with mm, and reinforcement was applied to hair and expressed the load with it with the numeric value (g) when cutting.

[0022] The example of formula 1 zirconyl-chloride ( $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ ) 0.5 weight section, the urea 5 weight section, the L-cystine 3.0 weight section, and the citric-acid 3.0 weight section were dissolved in purified water, the whole quantity was made into the 100 weight sections, and A liquid was prepared by adding monoethanolamine until it is set to pH10.0. B liquid was prepared by dissolving the polyoxyethylene oleyl ether 1.0 weight section and the dried-sodium-sulfite 2.0 weight section in purified water independently, and making the whole quantity into the 100 weight sections. Subsequently, the first processing agent of pH9.5 was obtained by mixing this A liquid and B liquid equivalent [ every ].

[0023] In the solution which dissolved the example of formula 2 zirconyl-chloride 0.5 weight section, the urea 10.0 weight section, and the citric-acid 3.0 weight section in the purified water 100 weight section, after adding a cystine so that it may become 0%, 0.5%, 1%, 3%, 5%, and 7% of concentration, monoethanolamine adjusted to pH10.0 and A liquid was prepared. B liquid was prepared by dissolving the polyoxyethylene oleyl ether 1.0 weight section and the dried-sodium-sulfite 2.0 weight section in purified water, and making the whole quantity into the 100 weight sections independently. Subsequently, first processing agent 6 kinds with which the cystine contents of pH9.5 differ were acquired by mixing this A liquid and B liquid equivalent [ every ].

[0024] Example of formula 3 cyanuric chloride is dissolved in ethanol, and five sorts of second processing agents, 0.1 % of the weight of concentration, 0.3 % of the weight, 0.5 % of the weight, 1.0 % of the weight, and 2.0 % of the weight, are prepared.

[0025] The second processing agent containing 2g (made in ICI, Inc., trade name pro Aster tataricus black PN) of black reactive dye, 5g of sodium sulfates, and 10g of ureas per 100ml of example of formula 4 water and with a dichloro thoriadiny group is prepared.

[0026] By the first processing agent obtained in the example 1 of the example formula of reference, the hair for a trial beforehand processed for 10 minutes at 55 degrees C is wound around a rod with a diameter of 6mm, and it dips in each of five sorts of these cyanuric chloride ethanol solutions, and processes for 10 minutes at 25 degrees C. Subsequently, after taking out these samples, it dips in a sodium-carbonate water solution 0.1%, and processes for 10 minutes at 25 degrees C, and a set is fixed. Thus, the hair which carried out set processing is rinsed, after being air-dry, that wave effectiveness is measured and this result is shown as a graph I of drawing 2. Moreover, when reinforcement was measured about each of these samples, it was the range of 5.8 - 6.0 g/d. Furthermore, except changing the processing temperature by the cyanuric chloride ethanol solution and the sodium-carbonate water solution into 40 degrees C, when set processing was performed by the same actuation as the above and the wave effectiveness was measured, the result shown in the graph II of drawing 2 was obtained. Moreover, the reinforcement of these samples was the range of 6.1 - 6.3 g/d.

[0027] After twisting the grizzled black hair wound around the rod around the first processing agent obtained in the example 1 of example 1 formula and processing for 20 minutes at 37 degrees C, a dipping is carried out to the processing agent for a hair set which is the above, and was made and prepared, and it processes for 10 minutes at 25 degrees C. Subsequently, 0.5g of sodium carbonates and 100ml are added into this processing agent, and it considers as the third processing agent, and processes for 10 more minutes at 25 degrees C. Thus, the hair which carried out set processing was rinsed, and when air-dry, the uniform hair dyed black was obtained. Next, when the wave effectiveness and reinforcement were measured, the number of the wave effectiveness was 3.5 and reinforcement was 5.8 g/d. Moreover, the hair uniform like the place which operated similarly and completely carried out set processing dyed black was obtained except changing the processing conditions of the first processing agent in 10 minutes at 55 degrees C. The number of the wave effectiveness of this thing was five, and reinforcement was 6.2 g/d.

[0028] After processing a grizzled hair bundle for 10 minutes for 20 minutes or at 55 degrees C by 37 degrees C using each first processing agent obtained in the example 2 of example 2 formula, wind this around a rod, it was made to curl, the dipping was carried out to the same processing agent for a hair set as an example 1, and set processing was carried out similarly. Thus, all obtained the uniform hair dyed black. It is shown in drawing 3 and drawing 4 by making relation of the cystine concentration in the first processing agent and the wave effectiveness in such hair, and relation between cystine concentration and reinforcement into a graph. As for the inside I of drawing, what was processed for 20 minutes at 37 degrees C, and II process for 10 minutes at 55 degrees C. As shown in these drawings, when performing set processing of hair using the processing agent of this invention, more excellent the wave effectiveness and reinforcement can be obtained by choosing the presentation of the first processing agent suitably.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] The state diagram of hair showing the evaluation phase of the wave effectiveness in this invention.

[Drawing 2] The graph which shows the relation between the active principle concentration in the processing agent for a hair set of this invention, and the wave effectiveness.

[Drawing 3] The graph which shows the cystine concentration in the first processing agent of the processing agent for a hair set of this invention, and the relation of the wave effectiveness.

[Drawing 4] The graph which shows the relation between the cystine concentration in the first processing agent of the hair set processing agent of this invention, and the reinforcement of processing hair.

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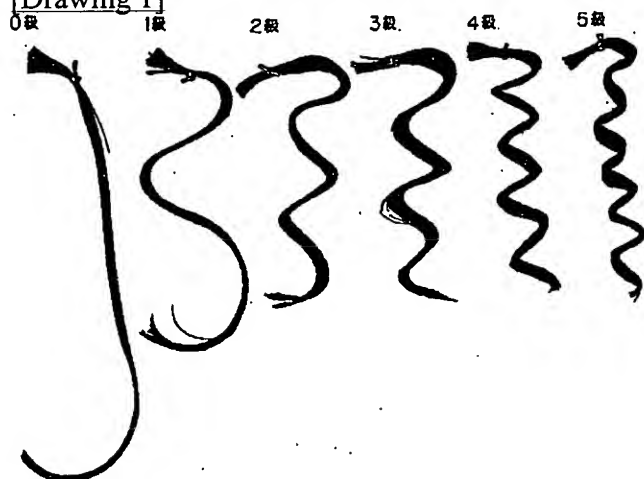
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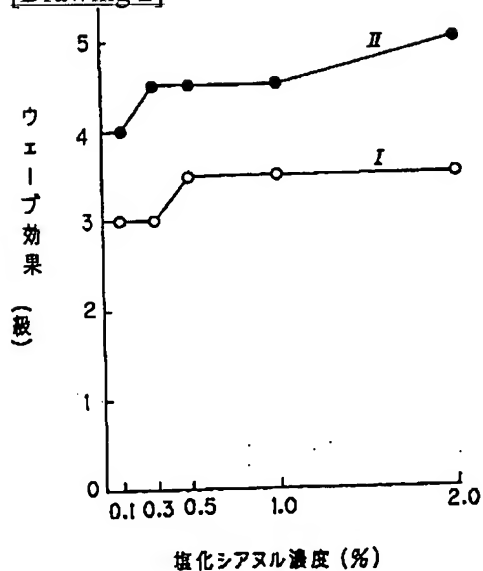
DRAWINGS

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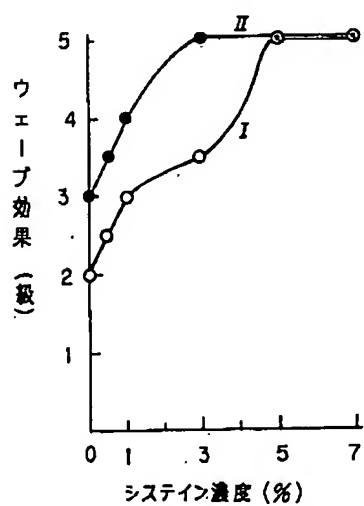
[Drawing 1]



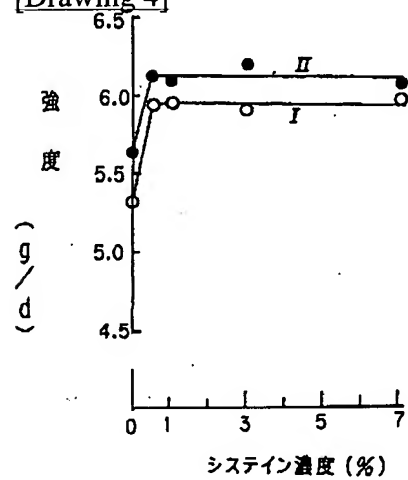
[Drawing 2]



[Drawing 3]



[Drawing 4]



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